Characterization of the new NSLS infrared microspectroscopy beamline U10B

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Abstract

The first of several new infrared beamlines, built on a modified bending magnet port of the NSLS VUV ring, is now operational for mid-infrared microspectroscopy. The port simultaneously delivers 40 mrad by 40 mrad to two separate beamlines and spectrometer endstations designated U10A and U10B. The latter is equipped with a scanning infrared microspectrometer. The combination of this instrument and high brightness synchrotron radiation makes diffraction-limited microspectroscopy practical. This paper describes the beamline's performance and presents quantitative information on the diffraction-limited resolution.

Keywords: Infrared microspectroscopy, infrared synchrotron radiation, infrared beamline, diffraction, spatial resolution.

Introduction

The advantages of synchrotron infrared radiation for microspectroscopy are now well-documented[1]. Its application has been demonstrated in studies of biological tissue[2] and cells[3], semiconductor materials[4], and geological specimens[5]. Other ongoing efforts include studies of interplanetary dust particles, forensics, corrosion, structural composites, and polymer laminates. To meet the increasing demand for measurement time, several new infrared beamlines have been constructed at the NSLS. Of these, the U10B beamline for infrared microspectroscopy began operating in August of 1998. This is the first of four infrared beamlines built on modified bending magnet ports of the NSLS VUV ring. The U10B port delivers 40mrad by 40mrad to a Spectra-Tech *Irμs™* scanning infrared microspectrometer. For the VUV ring (bending radius = 191 cm), this angular aperture meets or exceeds the natural opening angle for infrared frequencies down to 250 cm^{-1} and thus delivers high brightness synchrotron radiation across the important mid-infrared, vibrational spectroscopy range[1].

In this paper we report initial performance results for this new beamline. With no apertures, the illuminated area at the sample is approximately 20 microns and the detector signal frequently exceeds the input limit of the spectrometer's electronics. The available brightness is sufficient for performing spectroscopy through a 3 μm diameter metal aperture to wavelengths approaching 10 μm. We have also tested the system's spatial resolution when the targeting apertures are set to a value less than the wavelength of light, and determined the width of the diffraction-limited spotsize at the sample location. It is anticipated that the beamline will be available to NSLS General Users early in the year 2000.

Beamline and Microscope description

The NSLS infrared beamlines intended for microspectroscopy are designed around an extraction system that collects ~ 40 mr from the VUV ring (bending radius ρ = 191 cm). Per horizontal milliradian, this opening angle collects essentially 100% of the infrared down to 250 cm^{-1} without degrading the brightness. Below 250 cm^{-1}, the collection efficiency decreases slowly with frequency, but continues to outperform thermal sources

well into the far infrared[6]. The extraction system is therefore more than adequate for the majority of infrared microspectroscopy measurements, which are performed at frequencies above 400 cm^{-1}.

Beam extraction is accomplished with a combination of plane and ellipsoidal mirrors that collect the infrared and focus it through a diamond window. This window separates the storage ring vacuum from the remaining optical components. A diagram of a typical infrared extraction system (but without the vacuum components) is shown in Figure 1. Assuming that the ellipsoidal mirror is optimally positioned, the focus becomes a ~1:1 reproduction of the original source, and serves as a new source point for the remaining optical systems. The infrared light from this focus is collimated and relayed to the interferometer of the IR microspectrometer. Off-axis parabolic mirrors are used for collimation, although spherical mirrors could be used without appreciable loss in source brightness[3]. The beamline's optical system has a modest number of optical elements, and the total optical distance from source to spectrometer endstation is about 6 meters. The collimated beam distance is less than half this distance, which helps to preserve peripheral regions of the extended source.

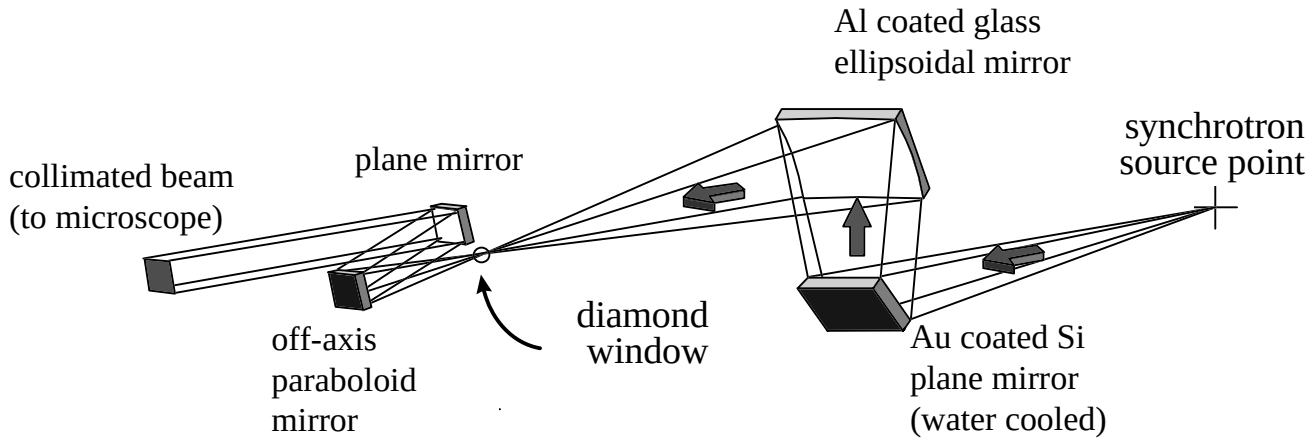

Figure 1. Schematic of synchrotron infrared beamline extraction optics. All components upstream of the diamond window are at ultra-high vacuum (UHV) (from Ref. 7).

The microspectrometer system at U10B is a Spectra-Tech Irμs™, provided by the Northrop Grumman Corp. as part of a collaborative research program. It consists of a standard Fourier Transform InfraRed (FTIR) spectrometer for the mid-infrared (400 cm^{-1} to ~ 5000 cm^{-1}), followed by a confocal microscope system and IR detector. A schematic of the instrument is shown in Figure 2. Details of the instrument's operation have been published elsewhere[1,7]. The actual microspectrometer installation is shown in Fig.3, with beamline components visible in the upper lefthand corner.

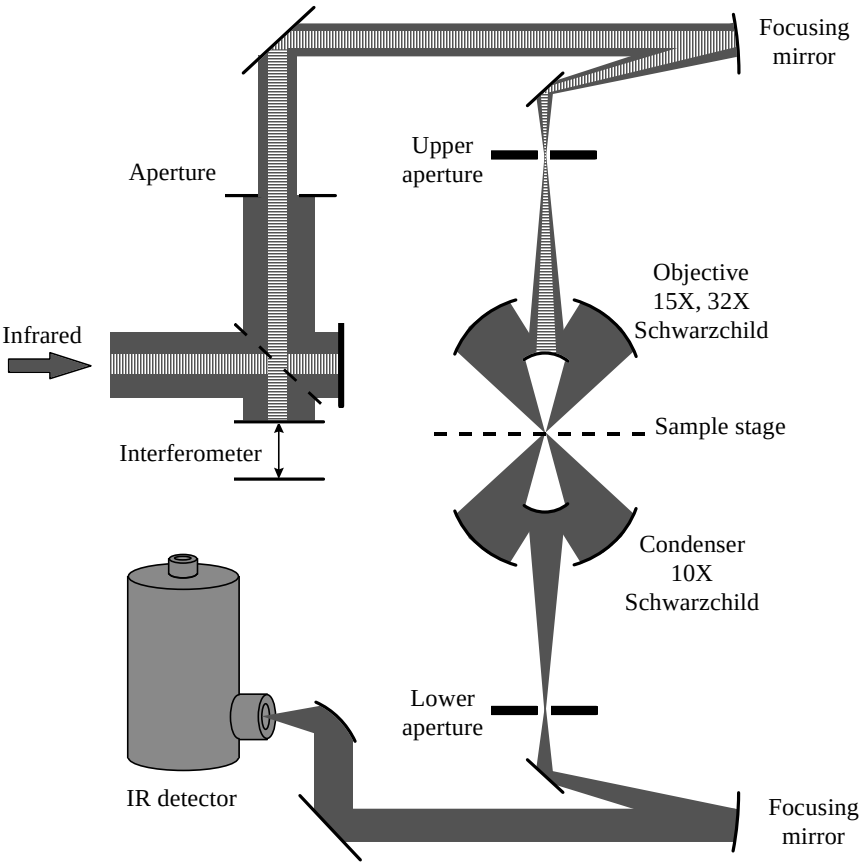

Figure 2. Schematic of the optical path inside the *Irμs™* infrared microspectrometer now installed at beamline U10B (from ref. X).

Performance

The system's performance was characterized in a number of ways. First, to ensure that the intrinsic source brightness was not degraded by the beamline optical alignment, the unapertured spotsize at the sample location was measured and compared to ray tracing predictions. The spotsize was determined by raster scanning a 3 μm diameter metal aperture across the sample focal plane and measuring the spectral signal at each position. The resulting pattern is oval in shape, with FWHM of 9 μm vertically and 16 μm horizontally. This is consistent with the ray tracing prediction for the extended synchrotron source. We also extracted the signal for two separate spectral ranges from the data: one for the 3 μm wavelength range, and the other for the 10 μm wavelength range. The results are shown in Fig. 4. While the horizontal extent of the source does not change, the vertical width grows with increasing wavelength. If the source were diffraction-limited in size, then the vertical size should increase as $\sim 0.8\rho^{1/3}\lambda^{2/3}$. Our vertical size shows less of an increase, as the electron beam dimensions are slightly larger than the diffraction limit at 3 μm wavelength.

The 3 μm pinhole, when located at the sample position, can be used to illustrate the high brightness capabilities of the synchrotron source. A standard method for quantifying the available S/N is to collect two spectra and calculate their ratio, with the deviations from 100% indicating the noise. We have produced a "100% line" from two spectra, each taken with 4 cm^{-1} spectral resolution and sampling times of ~2 minutes. The resulting ratio, shown in Fig. 5, deviates from 100% by less than 1% over most of the spectral range, and the local noise (see inset) can be smaller by a factor of two.

Figure 3. Photograph of the *Irμs™* scanning infrared microspectrometer installation at beamline U10B. The rectangular vacuum tank containing the collimating optics, along with the beam pipe, is visible in the upper lefthand corner. The storage ring is immediately to the left, behind a shield wall (not visible).

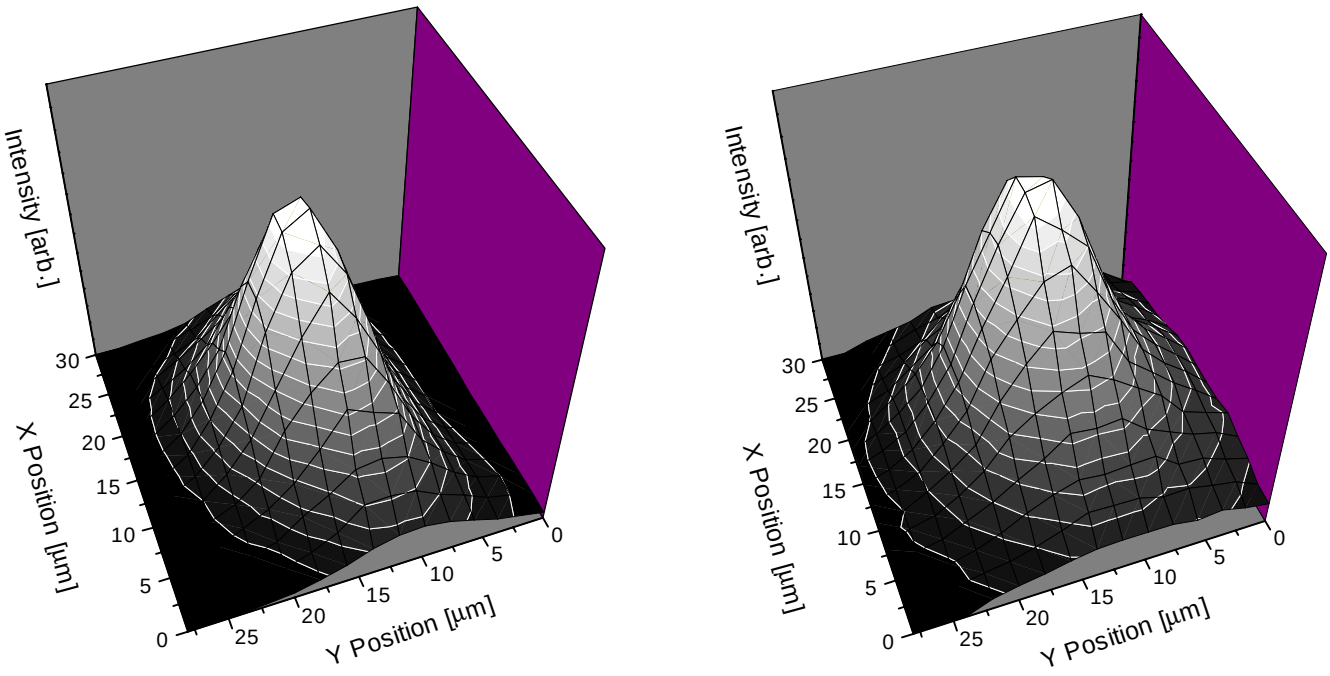

Figure 4. Illuminated spot profiles produced by the 32X objective (no apertures) at the sample location. *Left:* Profile for wavelengths near 3 μm. *Right:* Profile for wavelengths near 10 μm.

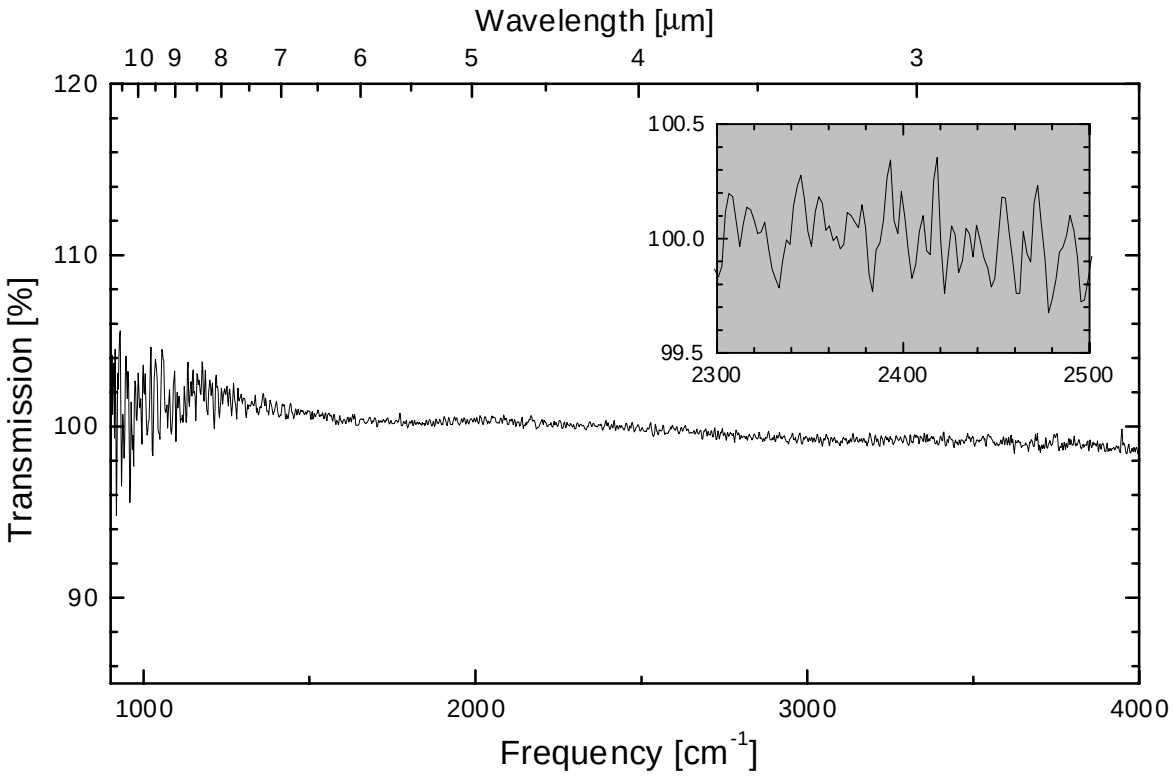

Figure 5. "100% line" for a true 3 μm diameter aperture located at the sample position. Two "single-beam" spectra were recorded at 4 cm^{-1} resolution and then ratioed. Acquisition time for each was about 2 minutes, and a signal-to-noise ratio of greater than 100 was achieved across most of the spectral range.

In practice, spatial resolution is achieved not by placing an aperture at the sample, but instead at an intermediate focus just before or after the sample focus. When used at a focus just before the specimen, this aperture serves effectively as a new source point. The spatial resolution is then determined by either the geometrical effective aperture size or diffraction, whichever is greater. As an example, we show two absorbance maps of a square-patterned section of photoresist on a BaF_2 substrate. The photoresist square is 12 μm on a side, and the absorbance is determined from transmission measurements. The *Irμs™* upper aperture was set to a 2 μm by 2 μm square, which is smaller than the wavelength for the entire spectral range of interest. Thus, diffraction will control the available spatial resolution. Absorbance features around 3 μm and 8 μm were used to produce the maps, which are shown in Figure 6. Note that the shape is much more clearly resolved at λ=3 μm than at 8 μm.

In general, we would expect the resolution to scale linearly with the wavelength. While the data shown in Figure 6 is suggestive of this, the quality is not sufficient to extract quantitative resolution information. To show the resolution capability more clearly, we have analyzed linear absorbance profiles across the edge of the same photoresist specimen. For this study, the instrument's upper aperture was set to a 1 μm wide by 30 μm long slit. The slit was oriented such that it was parallel to the photoresist edge. Spectra were collected at 1 μm increments, starting more than 20 μm before the edge and continuing 20 μm into the photoresist. The strength of the same two spectral features at 3 μm and 8 μm wavelengths were used to produce profiles of the step edge. These are shown in Figure 7. Again, since the aperture was set to a value substantially smaller than the wavelength of light, diffraction dominates the spatial resolution in all cases. We adopt a criteria commonly used in electrical engineering for the risetime of a step edge, namely the time (or in our case distance) for the "signal" to pass from the 10% to 90% signal level, and find that the spatial resolution is very close to the wavelength itself.

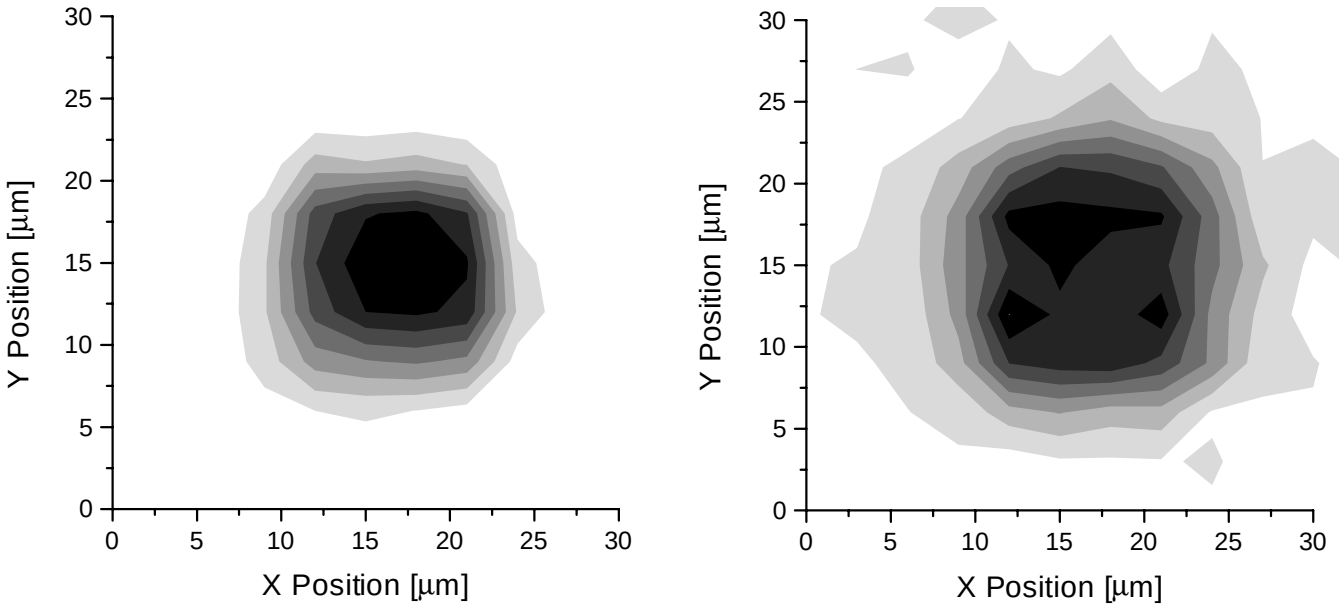

Figure 6. Absorbance maps for a 12 µm square patterned photoresist, showing the decrease in resolution with increasing wavelength as a consequence of diffraction. Darker shades represent greater absorption. *Left:* Absorbance at 3 µm wavelength (-NH). *Right:* Absorbance at 8 µm wavelength.

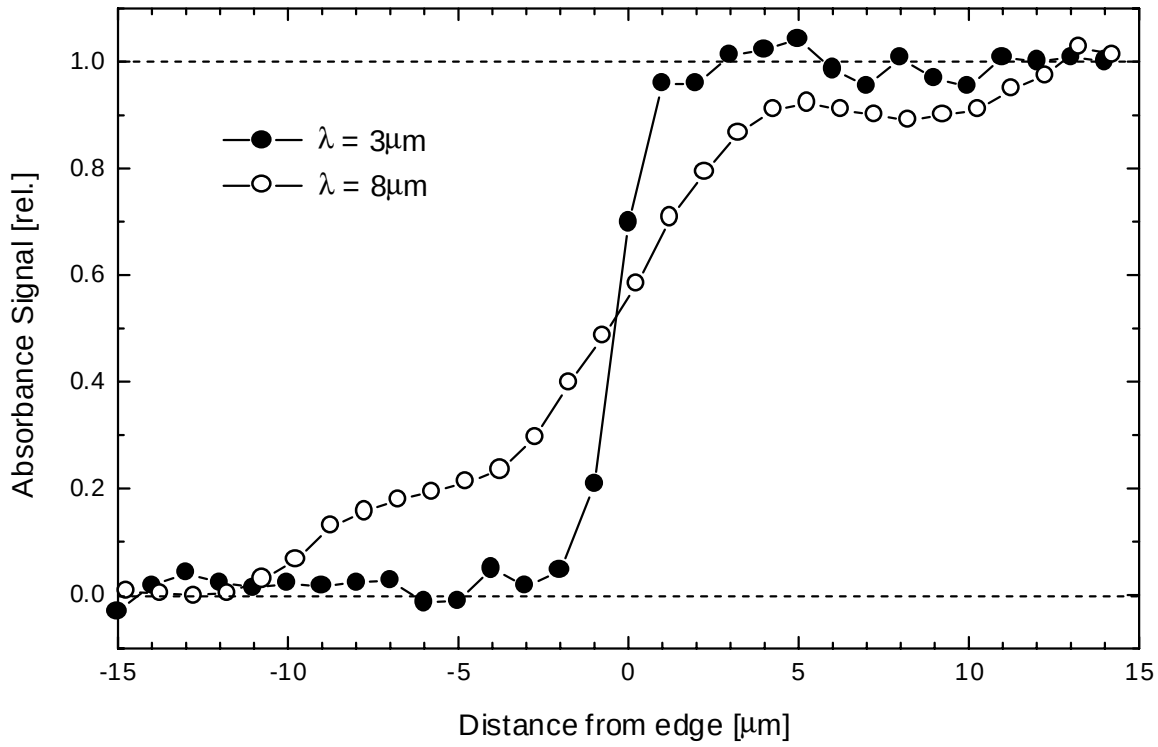

Figure 7. Absorbance profile from a linear transmission scan, passing from bare substrate (BaF_2) onto a thin layer of photoresist, showing the diffraction-limited resolution for 3 µm and 8 µm wavelengths. Using a "10% to 90%" criteria, the resolution is approximately equal to the wavelength. The upper aperture was set to a long, narrow slit (effectively 1µm wide). No lower aperture was used.

Summary

We have tested the performance of an infrared microspectrometer at one of the new NSLS infrared beamlines built on a 40 mrad by 40 mrad port. The spotsize produced at the instrument's sample location is in good agreement with optical ray tracing predictions, indicating that the beamline's optical system is not degrading the synchrotron source's intrinsically high brightness. This is confirmed by the quality of a 100% line acquired using a 3 μm diameter metal pinhole as a mask at the sample position. We also show that the unapertured spot profile at the sample location varies slightly with wavelength, as expected. Lastly, a practical spatial resolution very close to the wavelength of light is demonstrated and quantified using a photoresist pattern.

Acknowledgments

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